

E. Wickham

TECHNICAL DATA AND RECOMMENDATIONS

(Determination And Reporting Of Total Particulate
Matter, Water In Total Particulate Matter,
And Nicotine In Cigarette Smoke)

PM3000953217

TECHNICAL DATA AND RECOMMENDATIONS

(Determination And Reporting Of Total Particulate Matter, Water In Total Particulate Matter, And Nicotine In Cigarette Smoke)

1. Introduction

The following data and statement of methods and procedures for the determination of Total Particulate Matter (TPM), water in total particulate matter, and total alkaloids as nicotine (hereinafter referred to as nicotine) represents the consensus of major laboratories of the cigarette industry with only minor exceptions as noted. It should be emphasized that these industry laboratories have long and extensive experience in cigarette testing. The recommendations which follow are based on this experience, and reflect the need to observe certain precautions if reliable test results are to be obtained.

In preparing these recommendations, the aim has been to obtain results which are reliable within 5% of the reported value at the 95% confidence level. This statement would be statistically interpreted to mean that the true value of total particulate matter for a given sample reported as 20 mg per cigarette would be in the range of 19.5 to 20.5 and that in so concluding the investigator would be right 95 times out

of a hundred. It can also be shown statistically that to achieve this precision for a given sample approximately 200 cigarettes must be smoked. Because data and suggestions on methodology are related to the sampling plan, and selection of subsamples, our recommendations on sampling are first offered.

2. Sampling

?
If the objective of a cigarette testing program is to demonstrate differences between brands, a sampling procedure must be established so that the data obtained fairly reflect the characteristics of the product as a whole. Note that individual cigarettes within a given brand, or groups of cigarettes as represented by a package or carton, may vary in terms of weight, uniformity of blend, age, moisture content, filter dimensions, burning rate, etc. All of these can affect the particulate matter and nicotine delivery of the cigarette to a greater or lesser degree.

Moreover, the period which normally elapses between time of manufacture and purchase by consumer, as well as conditions of storage during that period, will produce in the market samples, variations from factory samples. If the

Commission is endeavoring to test what the consumer is getting, it seems clear that it must pick up what the consumer buys, i.e., cigarettes in the marketplace.

Reliable characterization of a brand as the consumer receives it, therefore, requires selection of the subsample to be smoked from a larger market sample population made up of cigarettes obtained from many locations.

The recommended procedure which follows is predicated on the pickup of 100 packages of cigarettes for each brand to be tested, from fifty market locations throughout the country. These can be selected by conventional statistical survey techniques. With some brands, supplementary pickup points may be necessary.

We realize of course that the 50 points suggested do not provide an adequate national random probability sample. For the time being, the Commission may well have to live with that inherent difficulty in its testing program which derives from time pressures and feasibility. We believe these pickup suggestions are practicable.

This pickup may be accomplished through an outside contractor, such as Western Union. The experience of several of the tobacco industry laboratories is that Western Union, originally proposed by us as an outside pickup contractor, can provide samples in good physical condition promptly.

Subsamples. The subsamples for smoking should be selected, as described below, from the one-hundred package sample which has been previously collected from fifty retail locations geographically distributed throughout the United States.

In using the procedure reported by Ogg (C. L. Ogg, J.A.O.A.C., 47, 356 (1964)), the participating laboratories were concerned with analytical techniques. Accordingly, the cigarettes which were smoked were not only from a specially prepared uniform sample but were also carefully weight-selected to minimize variations in cigarette weight. However, the consumer does not weight-select the individual cigarette he smokes. He removes each cigarette from the pack at random and smokes it. In order to duplicate this important aspect of consumer smoking, it is urged that weighing of cigarettes not be made. This is a necessary change in the procedure reported by Ogg.

The packages are carefully opened and three cigarettes are randomly selected from each package. These cigarettes are pooled and now represent a three hundred cigarette pooled subsample which after conditioning will supply the 200 cigarettes needed for smoking. (Comments on conditioning procedure are given below.)

As cigarettes are required for smoking, five cigarettes are randomly selected from the conditioned, pooled subsample, and are combined to provide a five-cigarette "port" sample. This procedure is repeated each time a five-cigarette "port" sample is required until the necessary number of "port" samples has been obtained.

In the selection of subsamples, cigarettes to be smoked should not contain soft spots nor be loosely packed or frayed at either end. In the final selection of cigarettes for smoking, damaged or defective cigarettes should be discarded and replaced with additional cigarettes drawn from the conditioned, pooled subsample.

In the light of what experience teaches are random inter-lab variations in the use of the Ogg method, the sample size to be smoked must be sufficient. As to the number of cigarettes to be smoked, the differences among types and brands that are to be measured, coupled with the technical difficulties and rigid controls required in the Ogg method, and the fact that the Commission is starting with a new laboratory and new equipment, demands that the number of samples smoked be adequate. Lost time on an inadequate sample is irrecoverable.

There need be no statistical cloud on the cardinal point that the Commission must sample often enough and get a big enough sample to secure precision. It must sample often enough to establish consistency. We suggest a minimum of

monthly sampling. Anything less plainly would be spotty, sporadic, and unreliable.

As a first approximation, the choice of at least forty as the number of "ports" to be smoked, and hence 200 cigarettes of each brand to be smoked is required in order to achieve results reliable within 5% of the reported value at 95% confidence. This calculation of 200 cigarettes is based upon our choice of an acceptable standard error as ± 0.5 mg. This selection is based on the assumption that TPM results will be reported to the nearest whole integer (1 mg) when reporting on a per cigarette basis.

The minimum of 200 cigarettes is also based upon a standard deviation of 1.6 mg per port which was the determined standard deviation among four different brands purchased once a week for three weeks and tested in the same laboratory under identical conditions. The choice of a standard deviation of 1.6 would be conservative since standard deviations among several major brands tested in a different laboratory over a six-month period have been observed on occasion as high as 2.8 mg per port. The detail of these observations are:

<u>Brand</u>	<u>Standard Deviation</u> mg/port
1	2.7
2	1.7
3	1.9
4	1.9
5	2.8
6	1.2
7	1.8
8	1.8

PM3000953223

Using the equation and the definitions given below, the number of cigarettes required can be calculated employing the conservative standard deviation of 1.6 mg per port.

From the Student's "t" test:

$$n = \frac{t^2 s^2}{E^2}$$

where n = the number of cigarettes smoked

t = the Student "t" value which is dependent upon the level of confidence desired. For 95% confidence it may be taken as 2.

s = the standard deviation

E = the maximum error which one wishes to tolerate with a given level of confidence.

Since the value for the mean standard deviation is based on one port (5 cigarettes), it is necessary to multiply by $\sqrt{5}$ to convert this to a per cigarette basis.

Thus:

$$n = \frac{t^2 s^2}{E^2}$$

n = the number of cigarettes smoked

$t = 2$

$s = 1.6 \times \sqrt{5}$

$E = 0.5$

$$n = \frac{(2)^2 (1.6 \times \sqrt{5})^2}{(0.5)^2}$$

$$n = \frac{(4) (12.8)}{0.25}$$

$n = 205$ cigarettes

To establish results for TPM to one-tenth of a milligram per cigarette with the same confidence, the number of cigarettes needed to be smoked gets very large indeed.

The calculation made above does not include allowance for inescapable between-laboratory variation. It has been suggested that the overall between-laboratory variations shown by Ogg in Table 3 are unrealistically large and that the corrected variations shown in Table 5 which eliminates data from four laboratories are more reasonable. We do not have at hand information which could support the exclusion of one-third of the laboratories in the Ogg work. We do know that in all analytical determinations with which we have had experience the between-laboratory variation is larger than the within-laboratory variations. Moreover, it is apparent that the within-lab standard deviations of Ogg on a uniform specially prepared sample, namely, 1.08 mg and 0.80 mg, are smaller than industry experience on multiple-brand testing, 1.6 mg and as high as 2.8 mg on occasion as indicated above. Thus, the within-laboratory standard deviations do not apply when testing market samples.

Despite the fact that the above calculations do not take into account many additional variations which must

be considered in trying to estimate a value which is representative of a brand as purchased in the marketplace, it is suggested that they be used as a basis for the minimum number of cigarettes to be smoked. It is believed that proper use of a monitor sample (described later) will reduce procedural variations; and in any event appropriate statistical evaluation of continuing samples will indicate the extent to which the sampling is adequate.

Any smaller sample to be smoked in the Commission laboratory than the suggested minimum of 200 cigarettes would be hazardous on presently available data and experience as to the Ogg method. Less than monthly pickup will yield only guesstimates and afford no reliable data as to variations or consistency.

Whatever may be the statistically appropriate extrapolations from the data in the reported tentative Ogg method and tables annexed to that report, acceptable mathematical precision necessary for the proposed testing project, yielding results to the nearest integer, on the basis of present knowledge, will require as a minimum the smoking of not less than 200 cigarettes. It is not feasible at this time, responsibly, to estimate whether experience in testing will require an increase or a decrease in that number.

7
0

Sample Conditioning. In the opinion of a large majority of the tobacco industry laboratories cigarettes should be conditioned prior to smoking. However, one major laboratory dissents from this opinion. This laboratory believes that when smoke analyses are made for the purpose of comparing brands sampled from the market, the cigarettes should, as far as possible, be smoked at the same moisture content which they had when purchased. On the other hand, those laboratories recommending conditioning feel that it is necessary because otherwise the moisture content of the cigarettes smoked will be a function of both the age of the cigarettes and the ambient conditions at which the cigarettes are stored not only in the wholesale and retail outlets but also while being held prior to analysis. This variability in moisture content will, in turn, affect the burning rate and therefore the nicotine and TPM delivery of the cigarettes.

If cigarettes are to be conditioned, they should be placed loosely upon wire mesh shelves in a space in which the temperature and relative humidity are adequately controlled. It is recommended that cigarettes be conditioned at $75 \pm 2^{\circ}\text{F}$ and $60 \pm 2\%$ relative humidity for a period of time sufficiently long to allow the cigarettes to come to moisture equilibrium with the atmosphere of the conditioning chamber. The exact time required will depend to a large

extent upon the rate of flow of conditioned air around the cigarettes. Once the conditioning facilities are operating, simple experimentation can establish the time required for cigarettes to attain a constant weight. This time should be adopted as a standard procedure provided that conditions such as air flow, cigarette loading, chamber geometry, etc. are not changed.

If the "port" sample is selected and/or prepared for smoking in a room which is not maintained at the same temperature and relative humidity at which they were conditioned, the cigarettes should be reconditioned before smoking.

Industry experience indicates that conditioning cigarettes in desiccators over constant humidity solutions is cumbersome, requires a very large number of desiccators and takes an inordinately long time for the cigarette to reach equilibrium.

Monitor Sample. It is common in analytical work to include check samples in order to be certain that the results obtained on unknowns are valid. This concept is especially important with smoke analytical procedures because experience, in various tobacco industry laboratories, has shown that there are unexplainable day-to-day variations in analytical results obtained on a single sample. To obtain valid smoke analytical results on diverse samples over a

period of time, it is essential to make daily checks on a carefully prepared monitor cigarette. These daily checks will indicate when variations attributable to instrumental failure or other abnormal conditions occur which would not otherwise be noted. In addition, the daily results for the monitor may be related to the average for this sample over a period of time and the results obtained on other samples may be corrected for the day-to-day variation in analytical results.

In the manufacture of a satisfactory monitor sample, extreme care should be used in blending the tobaccos, in the selection of uniform cigarette paper and filters and in fabrication.

The monitor cigarettes should be conditioned in accordance with the procedures described above. The number of monitor samples which should be smoked daily will depend upon the uniformity of the particular sample, the degree of control over smoking variables and the accuracy desired. This number can only be determined by experience and statistical considerations.

There are many details associated with the production and use of a monitor sample which are beyond the scope of this discussion. Additional information and assistance

in the production of a satisfactory monitor sample will be provided if desired.

3. Operating Procedures

Smoking Machine Characteristics. Each laboratory of the industry has done considerable work on development of analytical procedures for determination of nicotine and TPM in mainstream smoke. In spite of this, as noted by Ogg, further work is believed to be necessary to obtain a totally satisfactory method.

Nonetheless for strictly analytical purposes the puff characteristics in the Ogg method are representative averages and, although arbitrarily chosen, are exactly defined. Several different smoking devices are being used in the industry to achieve these puff characteristics.

Essential to the testing procedure is a smoking machine that can do the job, produce accurate and consistent results, and permit true comparisons. The automatic smoking machine which is selected should be capable of achieving with all types of cigarettes the following puff characteristics, which are the basic conditions specified in the Ogg procedure:

Puff Volume. 35 ± 0.5 ml measured as volume

of smoke that will be drawn from the butt end of the cigarette under actual machine smoking conditions. Puff volume should be checked before and after each analytical smoking run with smoke collection trap in system.

Puff volume should be measured using a soap bubble manometer.

Puff Duration. 2 ± 0.2 sec. measured at the cigarette under actual machine smoking conditions by a soap bubble manometer and a suitable timing device.

Puff Frequency. 1 puff per 60 ± 1 sec.

Experience has shown that smoking machines differ widely in the ease with which these conditions can be achieved. It is absolutely essential to be certain that the machine be capable of achieving these conditions for a given cigarette.

Experience demonstrates that the automatic smoking machine utilizing a falling water column must be carefully and constantly recalibrated in order to accommodate successive smokings of cigarettes with widely different resistances to draw, or erroneous results will be obtained. We stand ready to afford specifications for the essential modification required and useful techniques for calibration.

Volume Between Filter Holder and Machine.

Volume between the filter holder and the puffing device should be kept to an absolute minimum.

Draft Control. The area in which the cigarettes are smoked should be free from extraneous drafts.

Cambridge Filter Assembly*

(a) Filter holder. A Lucite (or aluminum) filter holder consisting of threaded inner and outer parts and a gasket of Teflon or other suitable materials.

(b) Filter disc. Cut discs 1.74" (44 mm) in diameter from CM113A fiber glass sheet made by the Cambridge Filter Corp., 738 Erie Blvd., East Syracuse 3, N.Y., or equivalent filter material. Filters must collect at least 99.9% of all particles 0.3 microns in diameter and over at a flow rate of 28 linear ft. per min., have a maximum pressure drop not exceeding 93 mm water at 28 ft. per min., and contain not more than 5% acrylic-type binder.

It is recommended that in ordering filter material specifications as given above should be clearly stated.

(c) Lucite guide. A Lucite guide is used to assist in placing the rubber membrane on the filter holder.

* For a detailed description of smoking apparatus see Wartman, W.B., Cogbill, E.C., and Harlow, E.S., Anal. Chem., 31, 1705 (1959).

(The holder, guide, and filter medium, CM113A, both in sheets and as 1.74" diameter discs, are obtainable from Phipps and Bird, Inc., Richmond, Va.)

(d) Rubber membrane. Cut a square piece of medium latex dental dam, approximately 3.5 x 3.5 cm. Place between two pieces of rubber tile, or other suitable material, and punch a hole 4-6 mm in diameter in the center of the sheet with a cork borer of appropriate size; the size of the hole depends on the circumference of the cigarettes. Latex rubber sleeves, 8 mm in diameter and 20 mm long, may also be used.

(e) Rubber "O" ring. An "O" ring, 3/8" i.d., may be made by slicing off a thin section of 3/8" i.d. rubber tubing or may be purchased from Linear, Inc., State Rd. & Levick St., Philadelphia, Pa.

4. Smoking Procedures.

Apparatus Assembly. The Lucite guide is used to place a rubber membrane on the filter holder by inserting the offset end of the Lucite guide through the hole in the rubber membrane and then into the entrance tube of the filter holder; holding the guide and the membrane firmly against the filter

holder, roll the rubber "O" ring over the guide and into position around the membrane and in the groove on the entrance tube. It may sometimes be necessary to center the aperture and adjust its diameter by manipulating the rubber membrane. Trim the excess rubber membrane with scissors. Fit a Cambridge filter pad into the filter holder with the rough side toward the port through which the cigarette is inserted. Position the gasket against the filter disc and screw in the Lucite plug securely against the gasket. Tighten with a special wire wrench with the ends fitted into two sockets on the back of the plug.

Wipe gently with a soft cloth or tissue and weigh the filter assembly to the nearest 0.2 mg. Connect the filter assembly to the smoking machine by a short piece of rubber or other suitable tubing with heavy enough wall so that the cigarette and filter assembly will be held in a horizontal position. Test the smoking apparatus and filter assembly for leaks.. Insert a cigarette through the hole in the rubber membrane until the end of the butt is approximately flush with the inner end of the holder tube, i.e., to a depth of about 7/16". Take care that the butt end does not come in

5mm

11mm

contact with the filter discs. Withdraw the cigarette slightly so that the lip of the orifice in the rubber membrane projects outward and forms a snug-fitting collar without crimping or pinching the cigarette. Occasionally, it may be necessary to shift the position of the cigarette slightly in or out, to insure that the collar surrounds the smooth portion of the cigarette and provides a leak-free seal.

Smoking. Light the cigarette at the beginning of the first puff (an electric coil lighter is suggested). Smoke each cigarette until the burning cone reaches the mark and record the number of puffs taken on each cigarette.

If the operator anticipates that the cone will reach the mark during a puff, he should use judgment whether to allow cigarettes to burn beyond the mark or to stop smoking it short of the mark. In the smoking of cigarettes, "overs" should be balanced with "unders" for each pad. After the last puff, let the cigarette remain in the holder, free-burning, until a few seconds before the next puff will be drawn by the machine, then quickly remove the butt from the holder and allow a clearing puff of air to draw in the smoke remaining in the entrance port of the assembly.

A total of 5 cigarettes is smoked through each filter pad as described.* Immediately after 5 cigarettes have been smoked, disconnect the filter assembly from the apparatus, wipe, and weight to the nearest 0.2 mg.

If a smoking machine with an automatic cut off is used it is unnecessary to balance "overs" and "unders" and there is no necessity for a clearing puff.

Record gain in weight of the filter assembly and save smoke samples for water and nicotine analysis.

In smoking care should be taken to smoke a different brand on each port of each machine, and in replicate smokings random selection should be made of ports for brands since some variability between ports may be expected even on the same smoking machine.

The weight of total particulate matter equals the gain in weight of the filter assembly. This weight representing the total particulate matter (wet) from five cigarettes is recorded in mgs.

A total of 40 smokings of 5 cigarettes each is to be made on each brand and the average yield per pad determined.

* On the expectation that appropriate standardized filter pads will be utilized, it appears on the basis of industry experience that no problem of variant results will exist when five cigarettes are smoked onto one pad, where such smoking is to no less than a 30 millimeter butt length. If cigarettes are smoked to a shorter butt length, it will be necessary in certain instances to smoke only four cigarettes to avoid overloading the pad. With four cigarettes, there will in some instances be a considerable loss of precision.

5. Butt Length

If all cigarettes are to be smoked to a constant butt length, it is recommended that this butt length be 30 mm (the length used by Ogg) or that it be 3 mm beyond the tipping paper (filter overwrap), whichever is the longer, and cigarettes selected for smoking should be marked at this point. If a fixed length of cigarette is to be smoked, e.g., 47 mm, the cigarettes selected for smoking as above described should be marked at this point before insertion into the holder.

The choice of a 30 mm butt length is based on the fact that the Commission has indicated that any standardized test to be applied to cigarette smoking will be essentially the "Ogg method." The Ogg procedure specified a butt length of 30 mm. This is a reasonable approximation of the average butt length for cigarettes smoked by Americans in contrast to the shorter average butt length for European smokers. If a constant butt length is to be used, no logical reason is seen to alter this part of the Ogg procedure. Since smokers do not normally smoke a cigarette into a filter overwrap and some cigarettes now available have a 30 mm overwrap, the recommendation is made that "this butt length be 30 mm.

(the length used by Ogg) or that it be 3 mm. beyond the tipping paper (filter over-wrap), whichever is the longer."

To our knowledge there is no adequate study, published or unpublished, of the U. S. population which shows an average butt length less than 30 mm. That some fraction of the smoking public smokes to less than a 30 mm butt length is obvious from published works documenting 30 mm as approximating the national average. But it must be remembered that in a standard method, experimental practicality dictates choices of certain arbitrary average conditions, and that this consideration has also dictated the specification of other smoking parameters such as average puff duration, average volume, and average frequency, each of which significantly influences smoke delivery. Therefore, it is essential to choose an average butt length. Pragmatically it should be recognized that 99% of cigarettes produced in the U. S. can be smoked to a uniform 30 mm butt length, whereas 90% of U. S. filter cigarettes cannot be smoked to a 23 mm butt without deviation from this specification. It is completely unscientific to adopt a standard in which the exception is the norm in 90% of filter cigarettes.

6. Determination of Water in Particulate Matter
and Calculation of Dry Particulate Matter

It is recommended that the test results be reported on a "dry" basis. The particulate matter collected by the Ogg Cambridge Filter Method contains a very significant proportion of water. The presence of variable quantities of this water adversely affects the precision of the determination and its inclusion could influence differences found between brands.

The water content of the TPM is influenced by a number of factors including type of cigarette, kind of filter, tobacco blend, width of cut, etc. Consequently, it may vary widely in different cigarettes. For most cigarettes the water content will range from 6½% to 13% of the particulate matter (See Appendices A and B). Also if a shorter butt length than the 30 mm prescribed in the Ogg method were to be used, the proportion of water in the TPM would become even greater.

Because of the variability in water content due to the above factors, the determination of water and correction for it actually improves the precision of the method.

Water may be determined on each pad using a gas chromatographic procedure similar to that described by

Schultz and Spears Tob. Science 10 (75-76) 1966/. The extract remaining from the water determination may be used for nicotine analysis as described below.

If it is desired, water may be determined separately, i.e., on additional smokings, using the Karl Fisher procedure essentially as described by Cogbill et al. Tobacco Science 3 (136-138) 1959/.

The average yield of water per filter pad is then subtracted from the average yield of total particulate matter (wet) to obtain the average TPM (dry) per pad. This value is used in calculating results as milligrams of dry particulate matter per puff by dividing by the average number of puffs per pad or as mgs of dry particulate matter per cigarette by dividing the average yield per pad by 5.

7. Nicotine

The apparatus and basic procedures described by Ogg for total alkaloids as nicotine are considered to be satisfactory. If water in TPM is to be determined by gas chromatography, all of the resulting solution and pad, or an aliquot of an extract from the pad, may be used instead of distilling the alkaloids from the total pad as in the

Ogg procedure. The size of aliquot required will depend in part on the alkaloid content of a particular sample and if this value is low, a determination using the Griffith still is preferred since the total distillate volume is one-half that of the modified Kjeldahl still and the concentration is therefore twice as large.

In order to keep the distilland volume as low as possible in the Griffith still procedure it is considered desirable to acidify the aliquot portion with a smaller volume of more concentrated acid. One ml of 1 N HCl is satisfactory. When working with extracts it is desirable to add the acidified extracts to the Griffith still through a 50 ml separatory funnel.

8. Reporting of Results

Cigarette smokers vary greatly in their manner of smoking -- puffing rate, volume, frequency, length of cigarette smoked, etc. Certainly not all smokers puff down to a fixed butt length. For many smokers, a more meaningful expression of smoke quantity than that of total quantity delivered per cigarette is the measure of average smoke concentration, that is, the particulate matter or nicotine per unit volume. In the above procedure a convenient

unit volume measurement is the standard "puff" so that the smoke concentration would be expressed as "milligrams per puff." This number would be derived by dividing the total delivery of the cigarette smoked by the number of puffs. Such a number for example, would be more meaningful to a person who only smokes half a cigarette. It is, therefore, recommended that particulate matter and nicotine values be reported both on a "per cigarette" and on a "per puff" basis. We also recommend that the nicotine values should be subtracted from the dry particulate matter, so as to avoid the double reporting of the nicotine.

The Commission would undoubtedly subject itself to justifiable scientific and public criticism if it selected only a "per cigarette" basis.

These considerations underlie our recommendation that both methods be used in reporting the testing results.

Finally, in reporting results only significant digits should be shown. The sampling and methodological errors in the recommended procedures only justify results rounded off to the extent indicated below: